Post-Test Analysis of a 10-Year Sodium Heat Pipe Life Test

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Abstract

High-temperature heat pipes are being evaluated for use in energy conversion applications such as fuel cells, gas turbine re-combustors, Stirling cycle heat sources; and with the resurgence of space nuclear power both as reactor heat removal elements and as radiator elements. Long operating life and reliable performance are critical requirements for these applications. Accordingly, long-term materials compatibility is being evaluated through the use of high-temperature life test heat pipes. Thermacore, Inc., has carried out a sodium heat pipe 10-year life test to establish long-term operating reliability. Sodium heat pipes have demonstrated favorable materials compatibility and heat transport characteristics at high operating temperatures in air over long time periods. A representative one-tenth segment Stirling Space Power Converter heat pipe with an Inconel 718 envelope and a stainless steel screen wick has operated for over 87,000 hr (10 years) at nearly 700 °C. These life test results have demonstrated the potential for high-temperature heat pipes to serve as reliable energy conversion system components for power applications that require long operating lifetime with high reliability. Detailed design specifications, operating history, and post-test analysis of the heat pipe and sodium working fluid are described. Lessons learned and future life test plans are also discussed.

Introduction

High-temperature heat pipes are being evaluated for use in energy conversion applications such as fuel cells, gas turbine re-combustors, and Stirling cycle heat sources. The resurgence of space nuclear power has created renewed interest in their use as reactor heat removal components and as radiator components. In the temperature range between 500 and 1000 °C, heat pipes can offer the favorable features of passive, reliable operation, effective thermal coupling between noncontacting fluid streams, and modest cost (Rosenfeld and Ernst, 1999). Long operating life and reliable performance are critical requirements for these applications. Reliability for space-based applications is particularly critical because component replacement is generally difficult or impossible.
Heat pipes are a key component under consideration for the higher power dynamic power systems. National Aeronautics and Space Administration (NASA) Glenn Research Center (GRC) has been involved in the development of dynamic power converters for both nuclear and solar power conversion systems. Of particular current interest are 1) the development of a Stirling Radioisotope Generator (SRG) as a possible high efficiency alternative to Radioisotope Thermoelectric Generators (RTGs) for deep space missions and unmanned Mars rovers and 2) higher power Brayton and possibly Stirling or Rankine power systems for nuclear electric propulsion and power applications. Thermal energy is generally supplied at a high temperature (>1000 K) to the power conversion system. High-temperature heat pipes are one option to efficiently transfer thermal energy from the nuclear reactor heat source to the dynamic power converter. Heat pipes may also be useful for transferring waste heat from the power converter to the radiator. Heat pipes and other passive two-phase technologies are currently being considered for the purpose of transferring heat from the power converter to the radiator with minimal temperature drop and to spread heat within the radiator panels isothermally.

Stirling heat engines are being developed for electrical power generation on manned and unmanned Earth orbital, planetary missions, and terrestrial applications for utility and remote power generation. Dish Stirling solar systems and nuclear reactor Stirling systems are two promising applications of Stirling engine technology. Sources of thermal energy used to drive the Stirling engine typically have nonuniform temperatures and heat fluxes. Liquid metal heat pipe receivers are often used as heat transformers to uniformly deliver thermal energy at high temperatures to the heater heads of these Stirling engines. The use of heat pipe receivers can greatly enhance system efficiency and potential life span.

One issue that needed to be addressed during the design phase of heat pipe receivers is the potential solubility corrosion of the heater head section by the liquid metal working fluid. Stainless steels and nickel-based superalloys are standard materials of construction for high-temperature heat pipes and heater heads operating in the temperature range 823 to 1073 K. At these operating temperatures, some components of these materials are appreciably soluble in working fluids such as sodium, potassium, and NaK. Over a typical life span of 7 to 10 years, essentially pure working fluid condensate will condense on the heater head surfaces. The condensate will leach the soluble components of the heater head material and transport them to the evaporator section of the heat pipe. When the working fluid is evaporated again, the soluble materials are precipitated and essentially pure working fluid is returned to the condenser section to leach more material. The condensation heat flux for a Stirling heater head is typically 20 to 25 W/cm². For a 33 percent efficient 25 kW, Stirling engine this corresponds to approximately 760,000 liters of sodium per year condensing on a heater head.

To establish long-term operating reliability for sodium heat pipes, long-duration sodium heat pipe life tests were performed. This paper describes the design and performance results from these tests, which collectively establish the capability of sodium heat pipes in applications requiring high reliability over long time durations.

**Nomenclature**

<table>
<thead>
<tr>
<th>Acronym</th>
<th>Definition</th>
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<tr>
<td>EDM</td>
<td>Electron discharge machining</td>
</tr>
<tr>
<td>GRC</td>
<td>Glenn Research Center</td>
</tr>
<tr>
<td>ICP–AES</td>
<td>Inductively coupled plasma atomic emission spectrometer</td>
</tr>
<tr>
<td>MTI</td>
<td>Mechanical Technology Incorporated</td>
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<tr>
<td>NASA</td>
<td>National Aeronautics and Space Administration</td>
</tr>
<tr>
<td>RTG</td>
<td>Radioisotope Thermoelectric Generators</td>
</tr>
<tr>
<td>SRG</td>
<td>Stirling Radioisotope Generator</td>
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<tr>
<td>WDS</td>
<td>Wavelength dispersive spectroscopy</td>
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One-Tenth Segment Stirling Space Power Heat Pipe Life Test: Test Article Description

A representative one-tenth segment Stirling Space Power Converter (SSPC) heat pipe with an Inconel 718 envelope and a stainless steel screen wick was operated for over 87,000 hr (10 years) at nearly 700 °C. Detailed design specifications, operating history, and post-test analysis are described for the sodium heat pipe.

Heat Pipe Design

GRC funded a dual phase program to investigate solubility corrosion and to develop coatings that would essentially eliminate the solubility corrosion potential. A complete description of the work performed and the conclusions reached can be found in the Final Report for Contract No. NAS3–26925 (Dussinger and Lindemuth, 1997). The final task of the program was to fabricate a one-tenth segment of the current SSPC, the Starfish heater head heat pipe, utilizing the coatings and coating processes developed during the program. This heat pipe would then be life tested for up to 10 years by Thermacore as Phase 3. Unfortunately, the heat pipe intended for life testing had several weld failures after charging and processing. Because this pipe was no longer available, NASA acquired the original one-tenth segment heat pipe from Mechanical Technology Incorporated (MTI) and provided it for testing in the Phase 3 effort. The one-tenth segment heat pipe, fabricated by Thermacore for the SSPC project under MTI Subcontract No. 003–05034, was returned to Thermacore for testing on this program. This heat pipe was not fabricated with the protective coating.

The Starfish heater head heat pipe is doughnut shaped with an inner diameter of 30 cm, an outer diameter of 90 cm, and a thickness of 10 cm. Internal to the doughnut are 50 radial fins with 38 one-millimeter-diameter gas passages in each fin. The helium working fluid in the converter flows through the gas passages while the sodium in the heat pipe condenses on the outside of each fin. The inner diameter of the annular heat pipe is attached to the outer diameter of the Starfish heater head. The one-tenth segment is a 36° slice of the overall heater head and heat pipe. The 38 small-diameter helium gas passages are replaced with five larger diameter passages to allow for installation of a gas-gap water-cooled calorimeter to remove and measure the heat flow. Several photographs of the one-tenth segment are shown in Figure 1. The design specifications for this heat pipe are given in Table 1.

![Figure 1.—Internal and external views of the tenth segment life test heat pipe.](image-url)
TABLE 1.—DESIGN SPECIFICATIONS FOR THE TENTH SEGMENT HEAT PIPE

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Specification</th>
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<tr>
<td>Operating temperature</td>
<td>1023 K (750°C)</td>
</tr>
<tr>
<td>Working fluid and fluid charge</td>
<td>100 g, High purity sodium</td>
</tr>
<tr>
<td>Calorimetric heat transport</td>
<td>4500 W</td>
</tr>
<tr>
<td>Condenser surface heat flux</td>
<td>20 W/cm²</td>
</tr>
<tr>
<td>Envelope material and fill tube material</td>
<td>In 718 envelope; 316L SS fill tube</td>
</tr>
<tr>
<td>Wick structure material</td>
<td>316L SS, two layers 100 mesh screen</td>
</tr>
<tr>
<td>Arteries</td>
<td>Four; 0.318 cm (0.125 in.) inner diameter</td>
</tr>
<tr>
<td>Artery material</td>
<td>316L SS, 325 mesh screen</td>
</tr>
<tr>
<td>Coating</td>
<td>None</td>
</tr>
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</table>

Life Test Supporting Equipment Design

A cart-mounted supporting equipment arrangement was used to complete the life test on the heat pipe. The heat pipe was heated primarily by radiation from 12 silicon carbide heating elements. In order to minimize heat loss, the heating elements were surrounded by a 9-in.-thick graded insulation package. The insulation package was formed to fit and support the heat pipe and calorimeter package. The power was controlled with a phase angle power controller in conjunction with a PID temperature controller. The silicon carbide heating elements were operated in series at a relatively low voltage—approximately 30 V. A 10-kVA stepdown transformer was used to reduce the primary 208 to 35 V.

The desired operating temperature was set on the temperature controller. The input signal to the temperature controller was the heat pipe vapor space temperature (type K thermocouple). The temperature controller sent a control signal to the phase angle power controller to increase or decrease power to maintain the heat pipe at the set point. The temperature controller also had a latching, over temperature alarm feature. In addition, the temperature controller had a second control feature that is being used to energize the hour meter when the temperature is within 5° of the set point.

The power that the heat pipe transfers is extracted and measured using a gas gap calorimeter. The gas gap calorimeter consists of small-diameter water tubes that are inserted into the heater head gas passage holes. The gap between the heater head holes and the water tubes outer diameter is filled with a mixture of helium and nitrogen gas. This arrangement results in the calorimeter water tube, a gas gap, and the heat pipe wall. The sodium vapor generated in the evaporator section of the heat pipe condenses on the Starfish heater head webs releasing its latent heat of vaporization. The thermal energy is transferred through the thickness of the heater head web by conduction. Next, the energy is transferred across the gas gap by radiation and by conduction through the gas. By controlling the fractions of nitrogen (low thermal conductivity) helium (high thermal conductivity) in the gap, the thermal conductance across the gap can be modulated. The heat is then transferred through the water tube wall by conduction and transferred by convection to the water flowing through the water tubes. The temperature rise of the water flowing through the tubes and the flow rate are used to calculate the heat pipe power throughput. The distilled water coolant is pumped from a tank under the test setup, through a 50-µm filter, and into the calorimeter. The coolant exiting the calorimeter then flows through two liquid-to-air heat exchangers, which are also mounted under the test setup. Heat is ultimately rejected to the room air.

Life Test Operating Results

The program goal was successfully reached through 10 years of operation at 700 °C. The test operations began in summer 1996 and were completed on June 1, 2010. A total of 87,783 hr (10 years) of operation at 700 °C were accomplished over this time period. The heat pipe continued to operate without apparent variation in thermal performance for the entire duration of the life test program. A complete description of the history of the life test is presented in a final report supplied to NASA, which also describes the shutdowns and required supporting equipment repairs (Rosenfeld, Minnerly, and Dyson 2011).
Post-Operation Analysis Results

This section of the paper describes the post-test analysis of the sodium and the heat pipe envelope. The goal of this task was to measure and evaluate changes to the sodium chemistry and envelope metallurgy incurred during the long-term operation of the heat pipe.

Post-Test Analysis of the Working Fluid Chemistry

At the conclusion of testing, a sodium sample was removed from the heat pipe for analysis. After removal from the support test stand, the two heat pipe fill tubes were torch heated to drive liquid sodium out of them. The heat pipe was then placed in an argon-filled glove box, and the fill tubes were cut off. A clean stainless steel vessel with a 0.25-in. connecting tube was welded onto one of the fill tubes on the heat pipe. A tube section with a vacuum valve was welded onto the other fill tube. The heat pipe was then heated, and about 10 g of sodium were pushed into the collection vessel under argon pressure. The collection vessel was then placed in an argon glove box, and a solution was prepared by dissolving 1 g in deionized water in a glass Pyrex beaker. The sodium hydroxide solution was then diluted to 500 ml. The resulting solution was acidified with nitric acid to a 2.0 pH, as determined by pH paper.

The aqueous sample was placed into a Nalgene bottle and sent to Lehigh Testing Laboratories for chemical analysis. The sodium solution was scanned from ~160 to ~800 nm with a Spectro CIROS, side-on plasma, inductively coupled plasma atomic emission spectrometer (ICP–AES) equipped with a CCD detector. A background blank of 2000 mg/L sodium solution from a traceable stock solution was also scanned; this sample was also acidified with nitric acid. Element peaks above the background scan were noted and a method was generated for these elements, which were then quantified using the same ICP–AES. Calibration curves for each of the elements determined were constructed using traceable standards, which included a matrix of 2000 mg/L sodium based upon the sample preparation specifications. A comparison of the sodium peaks (standard, blank, and sample) suggests that the value has good integrity. Thus, a sample concentration of 1 g/500 ml or 2000 mg/L was used to quantify the elemental concentration data. In other words, the solution concentration in parts per million is equal to the concentrations of other elements found in the sodium sample. The following elements were found using this analysis method: 18 ppm lithium, 713 ppm boron, 15 ppm magnesium, 286 ppm aluminum, 7171 ppm silicon, 177 ppm potassium, 86 ppm calcium, 39 ppm chromium, 4 ppm manganese, 198 ppm iron, 98 ppm copper, 78 ppm zinc, 0.6 ppm strontium, 5 ppm zirconium, and 949 ppm barium.

The possible sources of these elements include being dissolved from the envelope or screen wicks, or that some were initially present as contaminants in the sodium fluid charge. Review of the Inconel alloy composition shows that boron, manganese, iron, chromium, silicon, and aluminum may have dissolved into the sodium from the envelope inner surface. Similarly, iron, chromium, manganese, and silicon are present as alloying elements in the 316 stainless steel screen wick material. All other elements found in the sodium sample could only have been present as contaminants in the initial sodium source, that is, in the original fluid charge. It appears that a representative sample of the original charge sodium may be available and a similar analysis on the source sodium will be performed to define the starting sodium chemistry.

Post-Test Analysis of the Envelope and Wick Materials

The inside of the heat pipe was neutralized and cleaned after recovering as much sodium as possible. Then several sections of the heat pipe envelope were selected for detailed analysis. The heat pipe was first cut into two pieces; smaller samples were then removed from the halves. A wire electron discharge machining (EDM) approach was used to remove the test samples. Figure 2 shows the two sections of the heat pipe after the samples were removed. The outer surface of the envelope retained a natural dark oxide coating.
A total of six envelope/wick samples were removed for analysis. Sample regions included envelope samples taken from the heat pipe evaporator, adiabatic, and condenser regions. For some locations, a single-wire EDM cut removed two samples that provided a lower side (evaporator) sample and an upper side (adiabatic) sample from each location. These samples were designated A and B, respectively. Table 2 describes the location of each sample. The samples were then delivered to GRC for metallography and electron microscopy analysis. Samples were sectioned, nickel-plated, mounted, and polished. Figure 3 shows the sample preparation steps. A detailed report on the analysis results is being supplied as a final report to NASA GRC (Rosenfeld, Minnerly, and Kosta, 2011). The following discussion presents one envelope-analysis example for each heat pipe region.
Heat Pipe Envelope

Electron microprobe microstructural analysis of the inner surface of the envelope in the heat pipe evaporator section showed about 30 µm that was significantly affected (Fig. 4). Significant porosity was found in the Inconel 718 surface that was in contact with the sodium. Figure 5 shows a microprobe wavelength dispersive spectroscopy (WDS) quantitative line scan of major elements found in test sample 1A near the sodium-exposed surface. In this 30 µm region, the relative quantity of nickel dropped substantially while some elements, including niobium, iron, silicon, and titanium, had relatively higher concentration than in the sample interior. Sample 5A showed similar results. Microprobe analysis also was performed on the outer envelope wall near the silicon carbide heaters. There appeared to be an increase in nickel concentration and a decrease in molybdenum and iron concentrations in a region about 100 µm in thickness. The elemental concentrations were constant throughout most of the wall thickness with no clear indications of a diffusion gradient.

Microprobe analysis was performed on samples taken from the adiabatic region of the heat pipe, for example Samples 1B and 5B, and from the condenser region, Sample 6. Analysis of these mounted and polished cross-sections showed similar metallurgy changes to those found in the evaporator region. An approximately 30-µm-thick region near the inner envelope surface had notable porosity, was depleted of nickel, and had increased concentrations of some other alloying elements.

Because the nickel was apparently removed from the inner envelope surface in the evaporator region, an effort was made to determine if nickel was transported by the sodium to other regions of the heat pipe. No evidence of nickel deposition was observed in any sample; in fact, nickel removal to a 30 µm depth was typically observed near the inner envelope surface in all of the analyzed samples. Loss of the nickel to the sodium would seem to be the most likely explanation; however the sodium chemical analysis did not reveal notable nickel levels. Plating out at cooler regions would also seem likely, but evidence of plating was not found. The higher concentration of nickel at the outer surface suggests possible diffusion through the wall, yet there was no concentration gradient indicative of diffusion. The outer surface of the heat pipe envelope was covered with a dark oxide that typically forms from long exposure to air at high temperature.
Figure 4.—Electron microprobe back scattered image showing the 30 μm affected region observed on the sodium side of sample 1A.

Figure 5.—Electron microprobe image and wavelength dispersive spectroscopy (WDS) quantitative line scan of major elements for test sample 1A near the sodium (inner) surface.
Heat Pipe Wick Structure Analysis

The screen wick material was analyzed to determine the effects of long-term exposure to the sodium working fluid. Observations of the electron beam welds indicated no evidence of corrosion in the weld metal or heat-affected zone. Electron beam weld penetrations were measured to be between 3.5 and 5.4 mm. Microprobe analysis indicated deposition of sodium, oxygen, and carbon on the surface of the screen wires. These elements were most likely residues of sodium hydroxide and sodium methoxide that remained from the sodium removal and disposal process. Some evidence was seen of species migration in the screen wires; this effect did not seem to affect the integrity of the wires. Physical observation showed that the wires retained ductility and did not separate from the envelope inner surfaces near the tack-weld locations.

Conclusions and Recommendations

A sodium heat pipe demonstrated favorable materials compatibility and heat transport characteristics at 700 °C while operating in air for over 10 years. A representative one-tenth segment Stirling Space Power Converter heat pipe with an Inconel 718 envelope and a stainless steel screen wick has operated for over 87,000 hr at nearly 700 °C. Post-test analysis revealed no significant degradation of the envelope or screen wick material as a result of the life test. The life test provides strong evidence for long-term chemical compatibility of sodium heat pipes at high operating temperatures.

Post-test analysis of the sodium working fluid showed small quantities of more than a dozen other elements, some of which may have been originally present in the sodium. An analysis is planned for the original sodium source, to determine the sodium chemistry changes due to its operational history as a heat pipe working fluid.

Significant migration of alloying elements was observed, though the total effect on the envelope integrity was minimal. A 30-µm region of the inner surface had significant porosity and nickel depletion. The cause of this material transport is still under investigation.

References

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**14. ABSTRACT**
High-temperature heat pipes are being evaluated for use in energy conversion applications such as fuel cells, gas turbine re-combustors, Stirling cycle heat sources; and with the resurgence of space nuclear power both as reactor heat removal elements and as radiator elements. Long operating life and reliable performance are critical requirements for these applications. Accordingly, long-term materials compatibility is being evaluated through the use of high-temperature life test heat pipes. Thermacore, Inc., has carried out a sodium heat pipe 10-year life test to establish long-term operating reliability. Sodium heat pipes have demonstrated favorable materials compatibility and heat transport characteristics at high operating temperatures in air over long time periods. A representative one-tenth segment Stirling Space Power Converter heat pipe with an Inconel 718 envelope and a stainless steel screen wick has operated for over 87,000 hr (10 years) at nearly 700 °C. These life test results have demonstrated the potential for high-temperature heat pipes to serve as reliable energy conversion system components for power applications that require long operating lifetime with high reliability. Detailed design specifications, operating history, and post-test analysis of the heat pipe and sodium working fluid are described. Lessons learned and future life test plans are also discussed.

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